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MINERALOGICAL COMPOSITION OF RESPIRABLE COAL MINE DUST

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by

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ABSTRACT

A methodology for the determination of the mineralogical compositions of coal mine dust is proposed. The analytical work is intended to determine the true weight percentages of the major minerals contained in airborne dust collected on filters by field personal sampling and fixed location sampling.

The techniques employ X-ray diffraction spectrometry and scanning electron and scanning transmission electron micro-analyses. Methods of specimen preparation and of the transfer of the dust samples from membrane filters to specimen holders are also described.

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COMPOSITION MINÉRALOGIQUE DE LA POUSSIÈRE INHALABLE DANS LES MINES

par

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RÉSUMÉ

Le présent document porte sur les techniques qui peuvent être utilisées pour déterminer la composition minéralogique de la poussière inhalable en milieu souterrain. La recherche analytique a pour but de déterminer les pourcentages en poids réel des principaux minéraux contenus dans la poussière en suspension dans l'air qui a été recueillie au moyen de filtres dans le cadre d'un échantillonnage manuel sur le terrain et d'un échantillonnage dans un endroit prédéterminé.

Les techniques nécessitent l'utilisation de la spectrométrie par diffraction des rayons X et de micro-analyses par balayage électronique et par balayage par transmission des électrons. Les méthodes de préparation des échantillons et de transfert de ceux-ci à partir des filtres de la membrane jusqu'aux récipients à échantillons sont également décrites.

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INTRODUCTION

The respirable dust research program of the Cape Breton Coal Research Laboratory (CBCRL) has as main purposes, an improvement of the understanding and the control of respirable dust in underground coal mines (1). The reduction of the risks of the development and progression of pulmonary disease in coal miners will require further knowledge of the constituents of respirable dust to which the miners are exposed.

A preliminary investigation by the Atlantic Coal Institute, using scanning electron microscopy, on the mineralogy of underground coal mine respirable dust has shown that the major minerals are illite, quartz, calcite, pyrite, kaolinite and siderite (2).

The use of federal government laboratories is proposed for various analytical methods to determine quantitatively the major mineralogical composition of coal mine dust samples collected by gravimetric sampling and prepared at CBCRL. Although the proposed methods are intended to comparatively investigate the true mineralogical composition of respirable coal mine dust, the feasibilty of extending the methodology to analyse samples routinely collected by industry in order to meet regulatory requirements will also be determined.

SAMPLES

During shift time dust sampling in underground coal mines, airborne dust samples are collected on filters of various types: Millipore (MP) mixed cellulose acetate and nitrate, Nuclepore polycarbonate, Whatman glass fibre, MSA polyvinyl chloride (PVC) and Selas silver membrane filters. Sampling systems consist of constant flow portable pumps, cyclones and filter holders. These samplers are used for fixed location sampling and personal sampling. The amount of dust collected on a filter varies from 0.35 mg to 7.15 mg depending on factors such as the filter types and sizes, the samplers used for dust collection, sampling duration, coal mining activity and work environmental condition.

X-RAY DIFFRACTION SPECTROMETRY

Coal mine dust samples collected on a silver membrane filter or pulverized mineral standard dispersed on the filter is analysed directly on an X-ray diffraction spectrometer (XRD) for mineral identification and quantification based on specific peak intensities. Those dust samples on MP filters are ashed by a low-temperature asher, dispersed in filtered water and filtered on silver membrane filters for the X-ray diffraction analysis.

SCANNING ELECTRON MICROSCOPY

Coal mine dust samples or mineral standards on a Nuclepore membrane filter are coated with carbon or gold film and observed directly by a scanning electron microscope (SEM) at magnification of 4000X and subsequently analysed by X-ray energy dispersive spectrometer (XED) for the major minerals in coal mine dust. Both single particle analysis and area scan are proposed. A Tracor Northern Software program is suitable for automatic point count by the XED system.

Dust samples collected on other types of filters, such as Millipore, Nuclepore and PVC membrane filters are low-temperature - ashed and transferred onto the Nuclepore filters for direct examination by a scanning electron microscope.

SCANNING TRANSMISSION ELECTRON MICROSCOPY

A scanning transmission electron microscope equipped with an X-ray energy dispersive system is suitable for analysing mineral particles below 10 μm in diameter (3). Other advantages over SEM and electron microprobe systems are higher morphological and X-ray spectrometrical resolution and capability of differential identification by electron diffraction in a transmission mode. Both micro-area scan and single particle analysis can be performed on the same specimen. Field collected coal mine dust samples on membrane filters are ashed in a low-temperature asher to remove carbon, organic compounds and filter material. The ash is then dispersed in

distilled water to form a suspension of a given concentration. Small amounts of this suspension were placed or filtered on coated electron microscope grids. The coating material can be carbon, fomvar or gold. The dried specimens are then ready for observation and micro-analysis.

If the sampling time can be adjusted to collect the sample on a Millipore membrane filter requiring an average number of particles per field of electron microscope observation, the direct sample transfer method can be applied. A sector of the loaded filter is cut and taped, dust side up, on a precleaned microscopic slide. This filter assembly is exposed to acetone vapour to produce a fused smooth surface on the collection side of the filter. This fused filter is then coated with gold or carbon to hold dust particulates by a thin coated film. Small segments of the filter of dimensions 2 mm x 2 mm are dissected out and placed with collection side up, on electron microscope copper grids. The grids are then placed on the top surface of a piece of polyurethane sponge which is almost immersed in acetone. The sponge provides a porous platform for the gentle chemical dissolution of the original membrane filter. When the filter material is completely dissolved (usually in 90 minutes), it leaves dust particles adhering to the coatings which are in turn supported by the grids. After drying in room temperature, specimens on the grids are ready for observation.

NUMBER OF PARTICLES AND SCAN AREAS TO BE EXAMINED

The working (or screen) magnification is proposed at 4000X. At this magnification, a particle 2 μm in diameter appears 8 mm in size on the electron microscope screen. Ideally, 500 particles or scan areas per sample are randomly selected for analysis. This can be done with an automatic counting software program in a Tracor Northern X-ray energy dispersive system. For manual work, however, it is proposed to reduce this number to 100 for practicality. In this case, a pair of specimens will be prepared from a sample as a cross-check for consistency. If a dust sample contains very few particles to give 100 particles for analysis, a minimum of 50 fields at this magnification will be required. The total area of these fields represents approximately 31,000 $(\mu m)^2$.

DETERMINATION OF WEIGHT PERCENTAGE FOR EACH MINERAL IN COAL MINE DUST

Quantitative analysis of mineralogical composition by the area scan method requires mineral standards of a respirable size and of a known proportion. For single particle analysis, measured particle sizes can be used to compute mass or weight of individual particles and thus percentage by weight of each mineral type can be evaluated by assuming the particle density.

CORRELATION BETWEEN RESULTS OBTAINED BY USING VARIOUS ANALYTICAL METHODS

The ultimate goal of this proposed investigation is to determine the true composition of minerals present in coal mine air to which miners are exposed. To achieve this goal, it is necessary: a) to improve the reproducibility of each proposed method, b) to compare and correlate the results of various analytical methods and c) to examine the consistency of weight percentages obtained by various methods when the same dust sample and standards are used.

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